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**MICROSTRUCTURAL EVOLUTION AND TENSILE
PROPERTIES OF SnAgCu MIXED WITH Sn-Pb SOLDER
ALLOYS (PREPRINT)**

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Microstructural evolution and tensile properties of SnAgCu mixed with Sn-Pb solder alloys

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Abstract: The effect of incorporating eutectic Sn-Pb solder with Sn-3.0Ag-0.5Cu (SAC 305) Pb-free solder on the microstructure and tensile properties of the mixed alloys was investigated. Alloys containing 100, 75, 50, 25, 20, 15, 10, 5 and 0 wt. % SAC 305, with the balance being Sn-37Pb eutectic solder alloy, were prepared and characterized. Optical and scanning electron microscopy were used to analyze the microstructures while “mini-tensile” test specimens were fabricated and tested to determine mechanical properties at the mm length scale, more closely matching that of solder joints. Microstructural analysis indicated that a Pb-rich phase formed and was uniformly distributed at the boundary between the Sn-rich grains or between the Sn-rich and the intermetallic compounds in the solder. Tensile results showed that mixing of the alloys resulted in an increase in both the yield and the ultimate tensile strength compared to the original solders, with the 50% SAC-50%Sn-Pb mixture having the highest measured strength. Initial investigations indicate the formation and distribution of a Pb-rich phase in the mixed solder alloys as the source of the strengthening mechanism.

Key words: Pb-free solder, Sn-Ag-Cu, Pb contamination, tensile properties, microstructure

INTRODUCTION

Increasing environmental and health concerns over the toxicity of Pb combined with the strict legislation on banning the use of Pb-based solders by the European Union have promoted widespread use of Pb-free solder alloys in the electronic industry.^[1,2] The Pb-free solder alloy Sn-3.0Ag-0.5Cu has been widely used as a replacement for traditional Sn-37Pb eutectic alloy.^[3] During the transition period from Sn-Pb to Pb-free solders, existing high reliability products with Sn-Pb joints that remain in the field likely have to be repaired with Pb-free alloys. Certain electronic assemblies that are excluded from the legislation, such as aerospace systems, may have to use Pb-free solders replacement parts due to the lack of available Sn-Pb electronic components. In both cases it is probable that Pb incorporation and mixing between Pb-free and Sn-Pb solder alloys will occur.

Correspondingly, a number of technical issues related to mixing Sn-Pb with Pb-free alloys need to be addressed. The influence of Pb contamination in Sn-Ag-Cu Pb-free solder alloy on the reliability of solder joints has been reported by Chung *et al*, showing that the solder joint can pass thermal cycling reliability testing and perform in an equivalent manner to Sn-37Pb eutectic solder joints.^[4] Thermodynamic analysis and the resulting microstructure of the reaction between Sn-Ag-Cu Pb-free and Sn-Pb solder alloys have been carried out experimentally^[5-8] and by simulation^[7-10] showing the formation of a low melting point Pb-rich phase through a eutectic reaction at 178 °C. The shear strength of solder joints with Pb contamination has also been tested by Zhu *et al*, showing no obvious difference at room but a degradation at higher temperature.^[6]

By whatever method, Sn-Pb solder joints repaired with Pb-free solder, Pb-free solder repaired with Pb-containing solder, or multiple reworking processes can produce a wide variety of mixed

solder compositions. The impact of mixing on the microstructural evolution of different amounts of Pb in Pb-free solders is unclear. In this work, the incorporation of different amounts of Sn-37Pb to Sn-3.0Ag-0.5Cu Pb-free alloys was studied. Microstructural and mechanical data of the mixed solder alloys has been measured and compared with the Sn-3.0Ag-0.5Cu Pb-free and Sn-37Pb eutectic solder alloys. The effect of strain rate during mechanical testing has been studied. Correlations between the evolution of the microstructure and mechanical testing properties are proposed.

EXPERIMENTAL

Fabrication of mixed solders was done by adding eutectic Sn-Pb solder alloy with a Pb-free solder. Commercial alloys of Sn-37Pb eutectic (SnPb) and Sn-3.0Ag-0.5Cu Pb-free (SAC) were used as starting materials and melted in the furnace at 400 °C for 3 hours to produce mixed solder alloys. In addition to the pure alloys, the compositions were SAC-5SnPb, SAC-10SnPb, SAC-15SnPb, SAC-20SnPb, SAC-25SnPb, SAC-50SnPb and SAC-75SnPb (all in weight percent). The molten solder in the crucible was chill cast in an aluminum mold to form cylindrical ingots 25 mm in diameter. Table 1 presents the chemical compositions of all solder alloys in which the compositions of the solder alloys were altered by varying the relative amount of the components (Pb-free and Sn-Pb alloys), with the Sn content making up the balance.

The evolution of microstructure with the Pb content in mixed solder alloys was studied using optical and scanning electron microscopy (SEM). Each sample was mechanically polished to a 0.05 μm final finish with colloidal silica suspension. Distribution of the different solder elements was determined using electron probe microanalysis (EPMA).

Mini-tensile tests have been performed with a computer controlled, custom built mini-tensile apparatus. Tensile specimens of 1.3 mm gage length and 1.0 mm gage width were electro-discharge machined from cylindrical ingots. These specimens were subsequently ground and mechanically polished to a 3 μm final finish with alumina suspension and to a thickness of about 0.7 mm. The small size of the dog-bone shaped sample was used in order to better approximate the small size and surface to volume ratio of solder joints. Tensile tests were conducted at room temperature using strain rates of 1×10^{-3} , 5×10^{-4} , 1×10^{-4} , and $5 \times 10^{-5} \text{ s}^{-1}$ to determine the effect of Pb content on the mechanical properties of mixed alloy as well as to determine the effect of strain rate on strength. Three specimens for each solder were tested. The fracture surfaces of the tensile specimens were investigated using SEM.

RESULTS AND DISCUSSION

Microstructural evolution of mixed solder alloys with the Pb content

Optical micrographs of the pure Sn-3.0Ag-0.5Cu-based Pb-free solder alloy and mixed solder with low Pb content, SnPb being from 5 to 20 wt% in solder alloys, are shown in Figure 1. The Sn-3.0Ag-0.5Cu Pb-free alloy exhibits dendritic β -Sn and ternary eutectic regions where Cu_6Sn_5 and Ag_3Sn intermetallic compounds (IMCs) were finely dispersed in the β -Sn matrix (Figure 1a). With the addition of Sn-Pb into the Sn-3.0Ag-0.5Cu Pb-free solder alloy, as shown in Figures 1b-e, the resultant solder alloys contain a segregated phase that appears dark or black in the interdendritic regions. Calculation of phase equilibria in a quaternary Sn-Ag-Cu-Pb system indicated it should be a Pb-rich phase.^[9] The microstructure of SAC-5SnPb mixed solder is shown in Figure 1b. Compared with the irregular and blocky shape of Cu_6Sn and Ag_3Sn precipitates in the eutectic regions of the pure Sn-3.0Ag-0.5Cu Pb-free alloy, most of the IMCs in the mixed solder alloys were in a spherical shape surrounded by a large β -Sn matrix. The Pb-

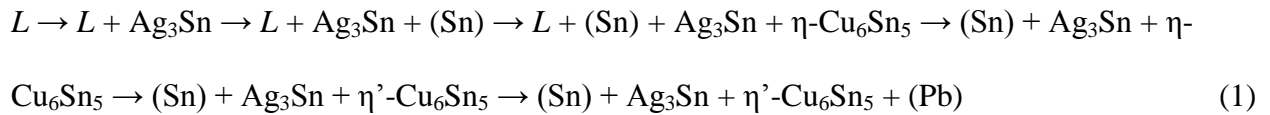
rich phase was uniformly distributed in a spherical shape with 1~2 μm diameters similar to Ag_3Sn or Cu_6Sn_5 IMCs. An increase in Sn-37Pb to 10, 15 and 20 wt% resulted in most of the Pb-rich phase having a spherical shape that predominately formed in the interdendritic regions in association with the IMC precipitates. It was also clear that increasing the Pb content in the mixed solder alloys led to a significant increase in the overall volume of the Pb-rich phase.

The microstructure of the Sn-37Pb eutectic alloy and mixed solder alloys in which the Sn-37Pb ranged from 25 to 75 wt % is shown in Figure 2. As shown in Figure 2d, at the eutectic composition Sn-37Pb solder was typically composed of binary Sn-rich and Pb-rich phases. In contrast to the low Pb content mixed alloys, in the SAC-25SnPb mixed solder alloy, the Pb-rich phase was still in the interdendritic regions but now the particles were no longer discrete but in a nearly continuous network along the grain boundaries (Figure 2a). For SAC-50SnPb solder, the Pb-rich phase was found to be in a continuous network with the Ag_3Sn and Cu_6Sn_5 near the eutectic phase along the boundary of the β -Sn phases (Figure 2b). With the content of Sn-37Pb in the mixed solder alloy at 75 wt%, as shown in Figure 2c, the Pb-rich phase was no longer just near the eutectic phase but had a bimodal distribution. Some of the Pb was near the eutectic phases in combination with the IMCs, similar to the other mixed solder alloys with a lower content of Sn-37Pb, while the other Pb-rich phase was located at the boundary of the β -Sn phases, similar to the Pb-rich phase in a Sn-37Pb solder.

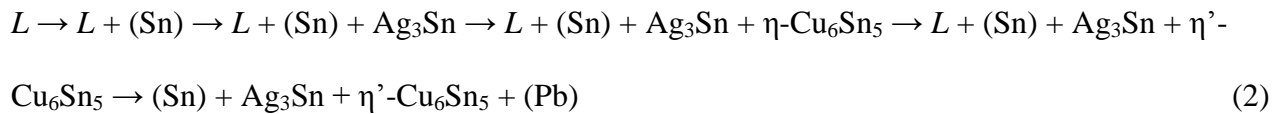
EMPA was performed to assess Sn, Ag, Cu and Pb distributions and the microstructural evolution in the mixed solder alloy matrix with different amounts of Sn-37Pb. Figures 3 and 4 are the results for one of the lower content Pb mixed alloys, SAC-10SnPb, and SAC-50SnPb with a higher Pb content, respectively. The Pb-rich phase precipitated in the interdendritic

regions along with Ag and Cu in both cases, but formed discontinuously with a spherical shape in the case of the SAC-10SnPb, while a virtually continuous network was observed in the SAC-50SnPb specimen. The chemical composition for the phases in the SAC-50SnPb was also investigated by EMPA. The compositions were 2.3 wt % Ag, 37.2 wt % Pb and 60.5 wt % Sn, in agreement with another traditional ternary eutectic alloy, Sn-36Pb-2Ag. This is a lower melting eutectic phase that formed from the last liquid to solidify during the ternary eutectic reaction at 178°C.

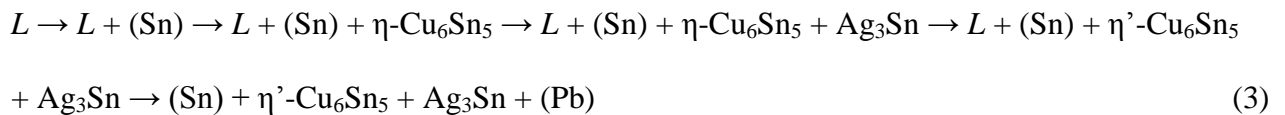
With the addition of different amounts of Sn-37Pb into Sn-3.0Ag-0.5Cu solder alloys, four kinds of phases, η -Cu₆Sn₅ or η' -Cu₆Sn₅, ϵ -Ag₃Sn, Sn-rich (Sn) and Pb-rich (Pb) formed during solidification. According to the SnAgCu-SnPb phase diagrams in the literatures ^[7, 9], the sequence of the precipitation of Pb phases has been changed by the amount of SnPb in the mixed solder alloys. If 5% SnPb is incorporated into SAC, the formation sequence during solidification from liquid (*L*) is:



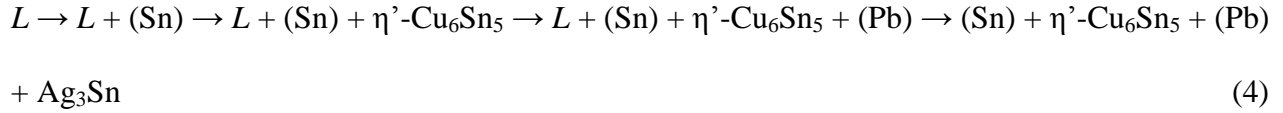
However, 10 % < SnPb < 50% is the composition of the mixed solder alloy then the phase formation sequence is:



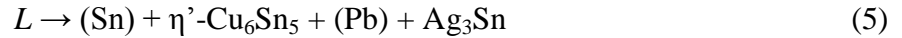
At 50 % SnPb content the solidification becomes



And finally at 75% SnPb and 25% SAC, the formation reaction is:



Also, there is a eutectic reaction that can occur at 178°C, which is below 183°C, the eutectic temperature for pure Sn-Pb:



With 5 wt% of Sn-37Pb in the mixture, Ag_3Sn phases were the first to precipitate from the liquid (reaction (1)), which was different from the other mixed compositions in which β -Sn was the first precipitated phase, resulting in the β -Sn grains difficult to observe in Figure 1b. Also, when the Pb phase forms is another difference caused by the amount of Pb in the mixed solder. With 5 wt% < SnPb \leq 50 wt% in the solder alloys, Pb phases did not form until the last step, after the precipitation of β -Sn, Ag_3Sn and Cu_6Sn_5 , and existed in the interdendritic regions. For the 75 wt% SnPb mixed solder alloy Pb precipitation happened before the eutectic reaction, and after the precipitation of β -Sn and Cu_6Sn_5 and along with Cu_6Sn_5 . Because the ratio for Pb to Cu in SAC-75SnPb is 27.75:0.125, and Cu seemed to be negligible to Pb, isolated Pb phases were produced in the solder alloy. Simultaneously, Pb precipitated with Cu_6Sn_5 and Ag_3Sn in the last stage for SAC-75SnPb, as was the case for solder alloys with small amounts of SnPb. Overall Pb precipitation that occurred before the eutectic reaction at 178°C led to isolated Pb phases, while Pb phases formed after the eutectic reaction where located in the interdendritic regions in the microstructure.

Effect of Pb content on the tensile properties

Typical tensile stress-strain curves of the mixed solder specimens at a constant strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ are shown in Figure 5. Averages of the yield strength (YS) and ultimate tensile strength (UTS) values from the mini-tensile tests are plotted in Figure 6 and presented in Table 2. Measured strengths for pure SnPb and pure SAC 305 matched handbook values, validating the mini-tensile test conditions. The addition of Sn-37Pb in Sn-3.0Ag-0.5Cu Pb-free solder alloy had a significant effect on the YS and UTS of mixed solder alloys. The UTS increased dramatically with the addition of Sn-37Pb from 5 to 20 wt%, as shown in Figure 5a. With up to 15 wt% SnPb addition, the UTS increased about 20% compared to that of Sn-Ag-Cu solder. At a SnPb amount of 25 wt% there was no significant increase in the UTS and YS, but the SAC-50SnPb mixed alloy, somewhat surprisingly, had the highest YS and UTS of the compositions tested. This strength was almost double the value of pure SnPb, and 50 % better than that of SAC solder. Continued additions of Sn-37Pb up to 75 wt% resulted in a decrease in the tensile properties.

The tensile results indicate that some strengthening mechanism occurred in the mixed solder alloys. The microstructural analysis indicated that the distribution of Pb phases changed with composition, and the most like reason for the increase in strength was the Pb-rich phases. Previous work on Sn-Ag-Cu Pb-free solder alloy found that the Ag_3Sn phases normally formed in a needle or plate-like structure, and had a higher strength than Sn-37Pb eutectic due to the existence of intermetallic phases ^[11], consistent with these testing results. In this work, for the lower Sn-Pb additions, the shape of the Ag_3Sn and Cu_6Sn_5 IMCs were smaller particles, as shown in Figure 1b. Also, the phase transformation sequence indicates that the Pb-rich phases were combined with Cu_6Sn_5 and Ag_3Sn to form the interdendritic phases. Pb-rich particles and IMCs may be attributed to the increase in tensile strength for the mixed solder alloys. With the

Sn-Pb content at 50 wt %, the eutectic network containing quaternary phases resulted in the highest strength. However, when the Sn-Pb content was 75 wt % in the mixed solder alloy, the precipitation of isolated Pb-rich phase along the β -Sn phases resulted in a decrease in the UTS and YS compared to the 50/50 mixture. However, the 75 wt% SnPb or 25 wt% SnPb in mixed solder alloy was still significantly stronger than either of the pure Sn-Pb and SAC305 alloys.

Effect of strain rate on the tensile properties

Mini-tensile specimens were also tested with different strain rates to determine how much the strain rate affected the testing results. The stress-elongation curves for pure Sn-Ag-Cu Pb-free and SAC-15SnPb mixed solder alloys shown in Figure 7 indicated that the strain does, in fact, have an effect on the mechanical properties of the solder alloys as the YS, UTS, and elongation at failure were dependent on the strain rate during testing. From the data it is apparent that as the strain rate decreased, the YS and UTS decreased, and elongation at failure decreased under all but one condition, but the overall response of the mixing alloys was unaffected by strain rates. Note that the mixed alloy remained significantly stronger than the pure Sn-Ag-Cu Pb-free under all test conditions. The tests on the other mixed solder alloys had a similar trend on the effect of strain rate as the SAC-15SnPb samples.

CONCLUSIONS

The microstructural evolution and tensile properties for the mixed solder alloys with different amounts of Sn-37Pb and Sn-3.0Ag-0.5Cu alloys were studied. With the addition of different amounts of Sn-37Pb into Sn-3.0Ag-0.5Cu solder alloys, Pb content resulted in a quaternary eutectic reaction, and led to the precipitation of a Pb-rich phase along with Cu_6Sn_5 and Ag_3Sn . In the mixed solder alloys with the content of Sn-37Pb from 5 to 50 wt %, the Pb-rich phase was

only located in the interdendritic region, and with an increasing amount of Sn-37Pb, the shape of Pb-rich and the intermetallic phases were changed from discontinuous spherical particles to a virtually continuous network. With a higher content of Sn-37Pb up to 75 wt % in mixed solder alloys, the Pb-rich phases were existed both in interdendritic regions combined with IMCs and in an isolated phase along the boundary of β -Sn grains.

The tensile testing results indicate that the strength of the alloys increases with mixing, with the SAC-50SnPb mixed alloy having the highest yield and ultimate tensile strength, almost double the value of pure SnPb. The fine dispersion of Ag_3Sn and Pb-rich precipitates in the matrix tends to strengthen the material. The effect of strain rates showed that as the strain rate decreased the yield, ultimate tensile strength and elongation at failure decreased.

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Table 1 Compositions of the solder alloys

| Name | Solders | | Compositon, wt % | | | |
|------------|-------------|--------------|------------------|------|-------|-------|
| | SAC wt % | SnPb wt % | Sn | Ag | Cu | Pb |
| SAC | 100 | 0 | 96.5 | 3 | 0.5 | 0 |
| SAC-5SnPb | 95 | 5 | 94.825 | 2.85 | 0.475 | 1.85 |
| SAC-10SnPb | 90 | 10 | 93.15 | 2.7 | 0.45 | 3.7 |
| SAC-15SnPb | 85 | 15 | 91.475 | 2.55 | 0.425 | 5.55 |
| SAC-20SnPb | 80 | 20 | 89.8 | 2.4 | 0.4 | 7.4 |
| SAC-25SnPb | 75 | 25 | 88.125 | 2.25 | 0.375 | 9.25 |
| SAC-50SnPb | 50 | 50 | 79.75 | 1.5 | 0.25 | 18.5 |
| SAC-75SnPb | 25 | 75 | 71.375 | 0.75 | 0.125 | 27.75 |
| SnPb | 0 | 100 | 63 | 0 | 0 | 37 |

Table 2 Mini-tensile results for the mixed solder alloys

| Compositions | $\sigma_{0.2}$, MPa | UTS, MPa |
|--------------|----------------------|----------|
| SAC | 38.9 | 41.6 |
| SAC-5SnPb | 39.7 | 42.9 |
| SAC-10SnPb | 44.7 | 48.2 |
| SAC-15SnPb | 46.7 | 49.7 |
| SAC-20SnPb | 46.1 | 48.7 |
| SAC-25SnPb | 45.8 | 48.7 |
| SAC-50SnPb | 52.3 | 59.4 |
| SAC-75SnPb | 46.8 | 54.0 |
| SnPb | 33 | 39.6 |

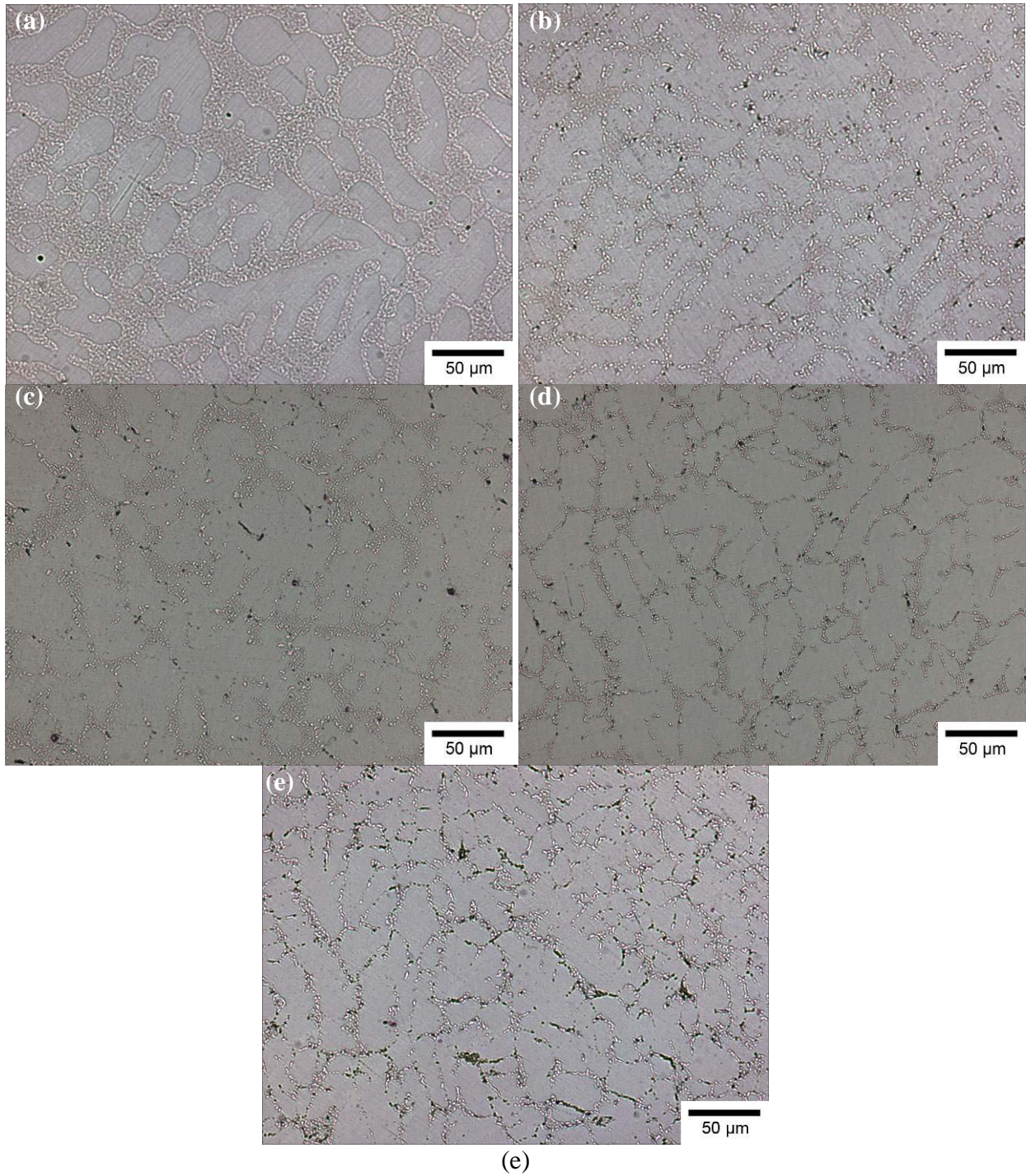


Figure 1 Optical micrographs of mixed solder alloys with lower content of SnPb
(a) Sn-3.0Ag-0.5Cu, (b) SAC-5SnPb, (c) SAC-10SnPb, (d) SAC-15SnPb, and (e) SAC-20SnPb

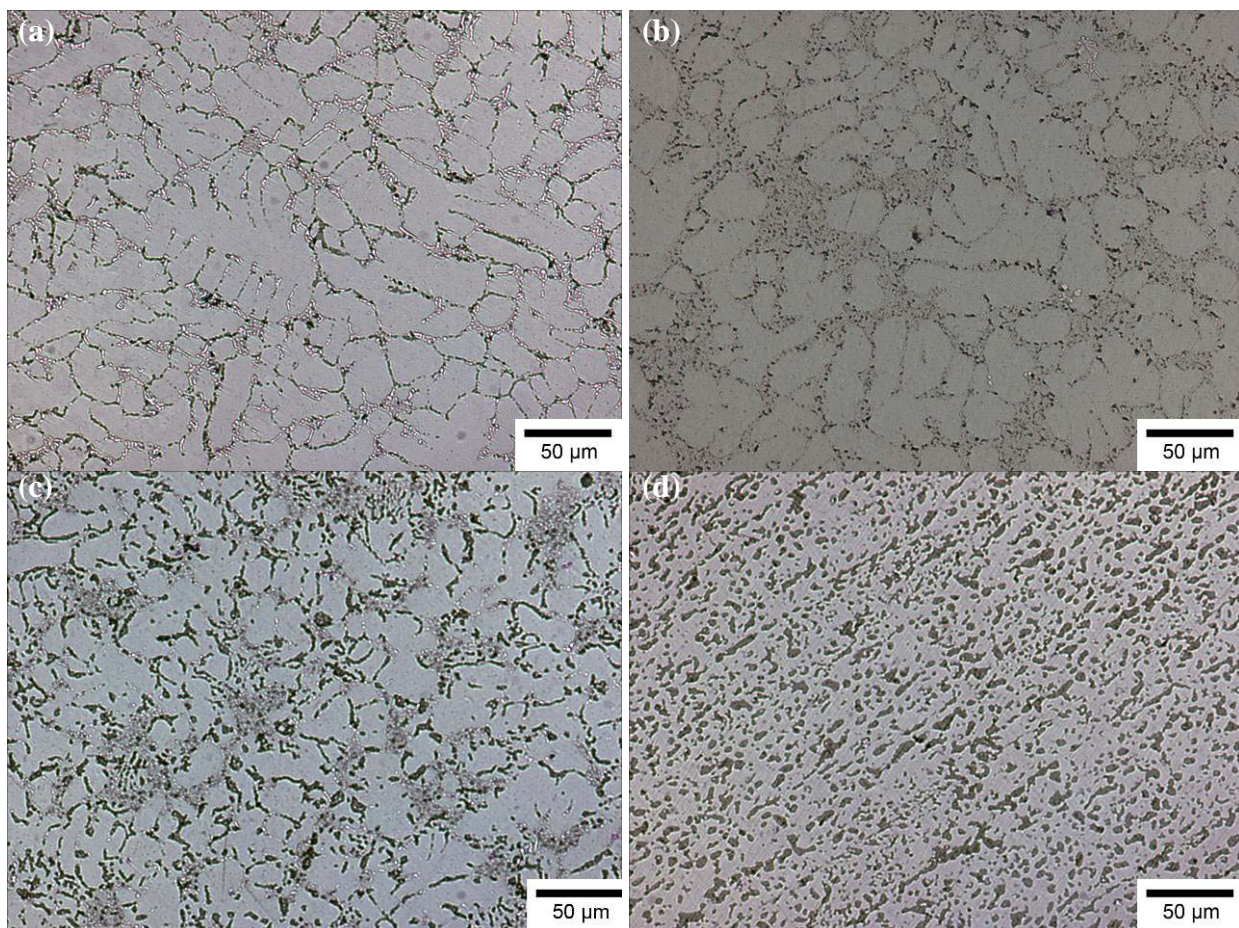


Figure 2 Optical micrographs of mixed solder alloys with higher content of SnPb
(a) SAC-25SnPb, (b) SAC-50SnPb, (c) SAC-75SnPb, and (d) Sn-37Pb

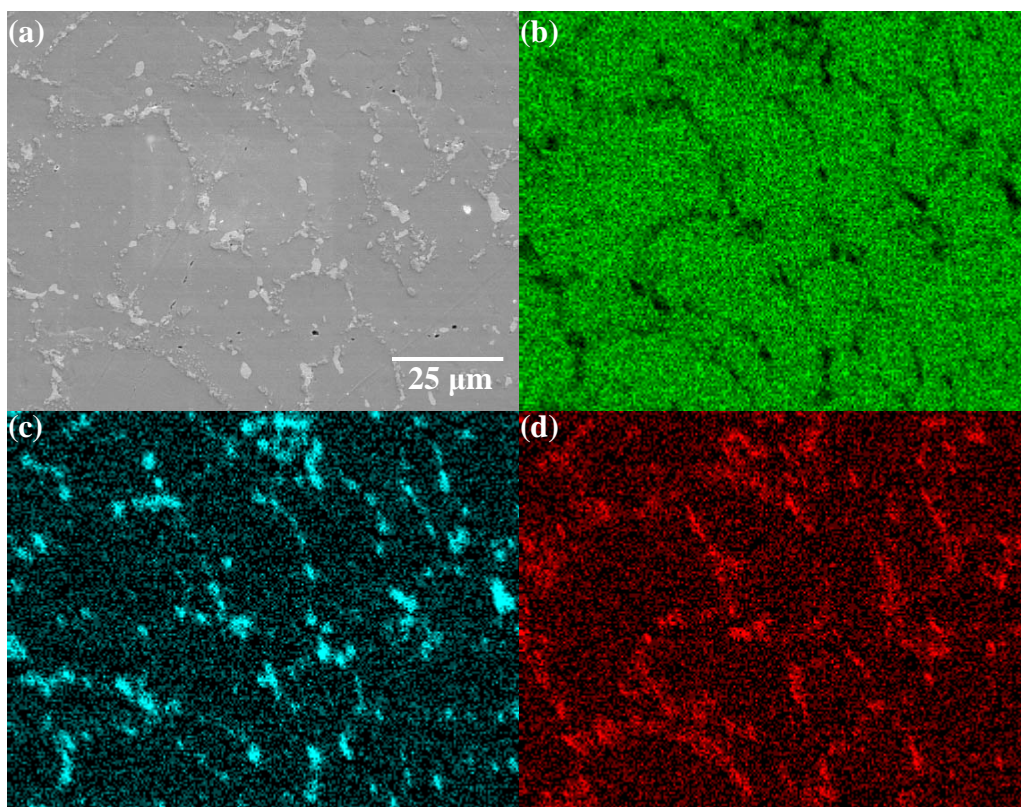


Figure 3 (a) SEM micrograph of low Pb SAC-10SnPb mixed solder and EDX element mapping for (b) Sn, (c) Pb, and (d) Ag

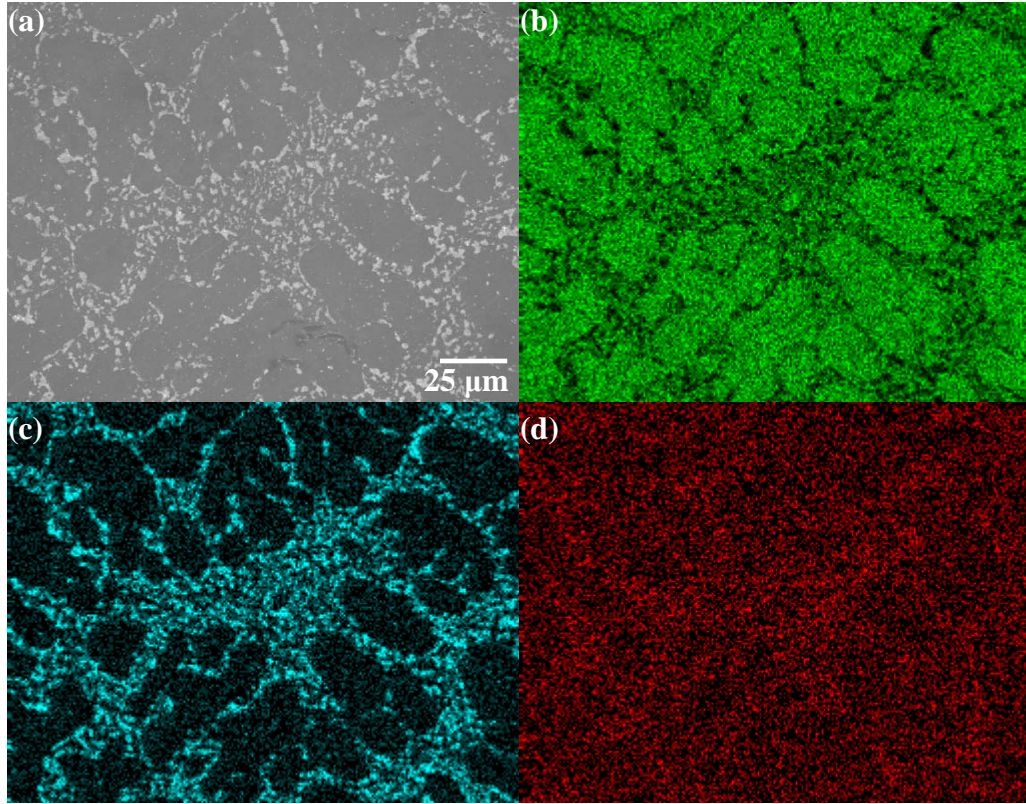
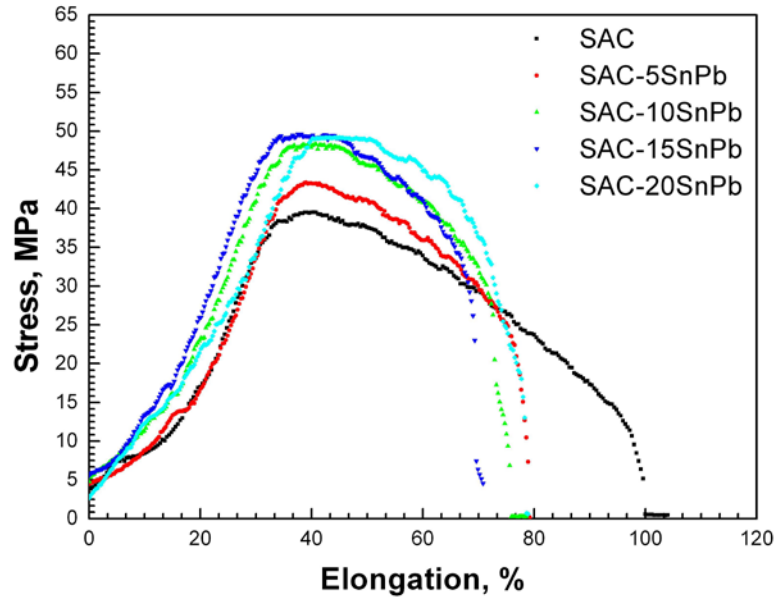
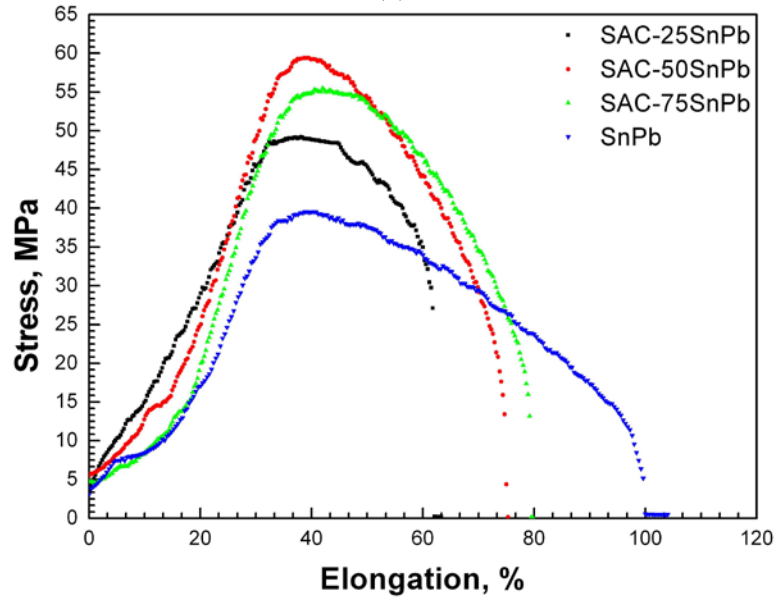


Figure 4 (a) SEM micrograph of high Pb SAC-50SnPb mixed solder and EDX element mapping for (b) Sn, (c) Pb, and (c) Ag



(a)



(b)

Figure 5 stress-strain curves for mixed solder alloys with lower content (a) and higher content (b) of Sn-37Pb in Sn-3.0Ag-0.5Cu Pb-free solder

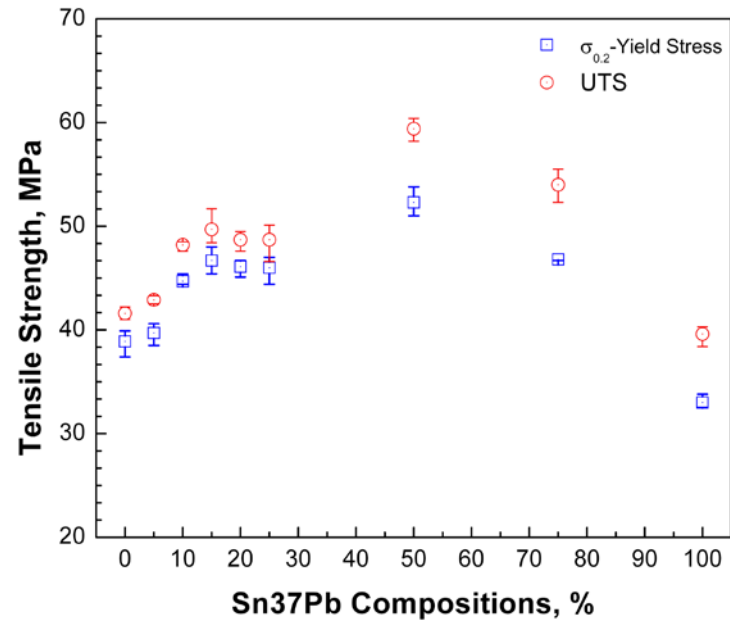


Figure 6 Effect of the content of Sn-37Pb in mixed solder on tensile strength

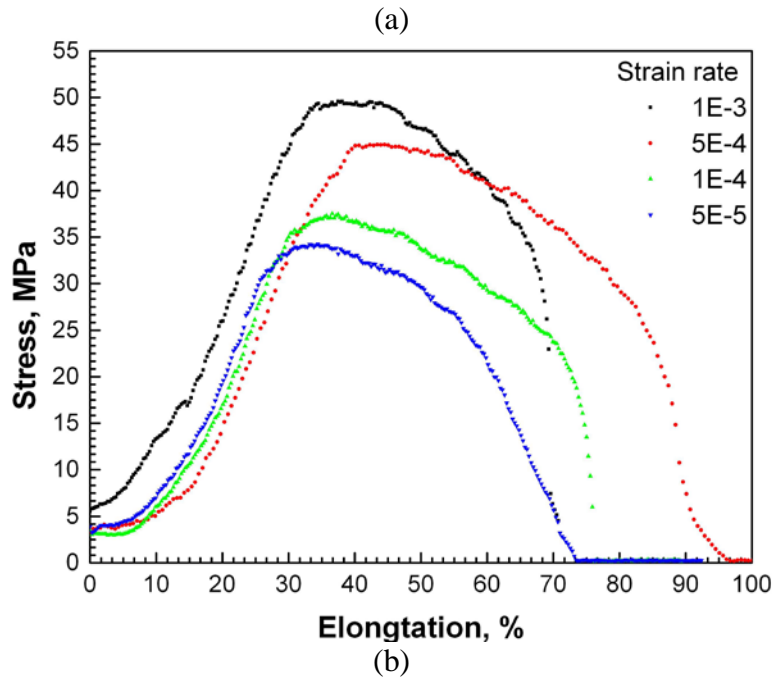
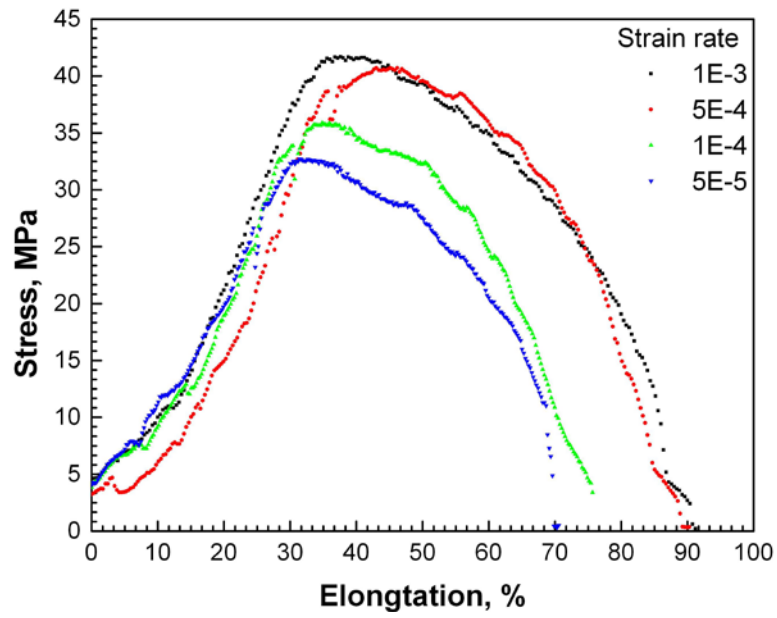


Figure 7 Stress-strain curves of (a) Sn-3.0Ag-0.5Cu Pb-free and (b) SAC-15SnPb mixed solder alloys at different strain rates